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ANALYTICAL REFERENCE SERVICE TRAINING PROGRAM. AIR SULFUR-DIOXIDE, NUMBER 1, JUNE 1963

Robert A. Taft Sanitary Engineering Center
Cincinnati, Ohio

June 1963

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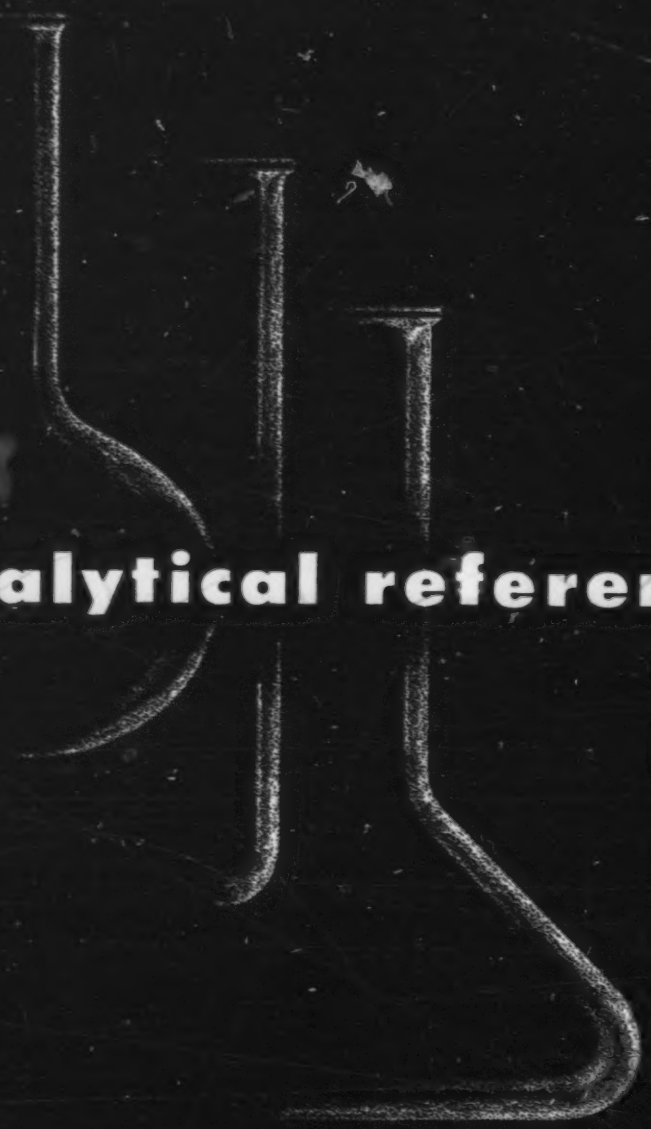

AIR SULFUR—DIOXIDE
NUMBER 1
JUNE, 1963

analytical reference service

TRAINING PROGRAM

Robert A. Taft

SANITARY ENGINEERING CENTER

U. S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE
PUBLIC HEALTH SERVICE
CINCINNATI OHIO

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Report

AIR SULFUR DIOXIDE NO. 1

A Study Conducted by the
Analytical Reference Service
TRAINING PROGRAM
for the
Division of Air Pollution

Public Health Service
Bureau of State Services

Robert A. Taft Sanitary Engineering Center
Cincinnati, Ohio
1963

58

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ACKNOWLEDGEMENT

The Analytical Reference Service Staff gratefully acknowledges the assistance of T. A. Hartlage, Chemist, Air Pollution Training, Training Program.

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ANALYTICAL REFERENCE SERVICE MEMBERSHIP

FOREWORD

The Analytical Reference Service is conducted by the Robert A. Taft Sanitary Engineering Center to provide a means of evaluating laboratory methods in the field of environmental sciences and engineering. Membership includes Federal, State, and municipal agencies, industries, universities, consulting firms, and foreign agencies. Cooperative studies by member organizations, through analysis of identical samples of environmental media and critical review of methodology, provide the mechanism for:

Statistical evaluation of procedures, including precision and accuracy.

Comparison of analytical procedures and results between laboratories having similar responsibilities.

Exchange of information regarding method weaknesses.

Improvement of existing methods or development of newer, more accurate methods to replace current procedures.

Development and evaluation of entirely new methodology for the determination of new pollutional components, such as pesticides, herbicides, antimony, cadmium, boron, selenium, phenol, and trace air pollution contaminants.

Initially directed toward examination of water, the studies have been broadened to include other media. Samples are designed and prepared to contain measured amounts of selected constituents. Decisions as to qualitative makeup are made by the staff of ARS, the membership, and consultants. Notice of a study is forwarded to each member with information regarding the qualitative makeup of the sample and special details regarding its handling.

A portion of the sample is shipped to each member who indicates a desire to participate in the study. Accompanying each sample are data forms which provide space for reporting numerical analytical values and a narrative critique of the procedures used. The laboratories are urged to comment on the methods, difficulties, modifications, sources of error, and other factors.

The results and comments of each study are compiled, as in the accompanying report. Experience indicates that a repeat study produces a sizeable increase in the number of participants. The first study designed to evaluate analytical methodology for the determination of pollutants in air was completed in 1958, with twenty-one participants. Forty-one agencies participated in the 1961 study, while 43 cooperated in this study.

A strontium-90 in water study has recently been completed and a report is being prepared. A study for the determination of surfactants in distilled water, sewage, and surface waters is being conducted by 100 members. The study to determine counting and identification of plankton in freshwater is being evaluated by some 60 members.

The selection of studies is guided by answers to the questionnaires distributed to member agencies to poll their interests. Results of this poll continue to serve as a useful aid in planning future studies.

Membership now consists of 165 agencies of diversified interests, with foreign interest increasing rapidly. The assistance and experience that agencies from other countries can bring to this program are invaluable to the entire membership.

H. P. Kramer
Chief, Training Program

STUDIES ON WHICH REPORTS HAVE BEEN COMPLETED

Water-Minerals	Calcium, magnesium, hardness, sulfate, chloride, alkalinity, nitrite, nitrate, sodium, and potassium. Studies completed in 1956, 1958, and 1961.
Water-Metals	Lead, copper, cadmium, aluminum, chromium, iron, manganese, and zinc. Studies completed in 1957 and 1962.
Water-Fluoride	Fluoride in the presence and absence of interferences, with and without distillation using a specified procedure. Studies completed in 1958 and 1961.
Water-Radioactivity	Gross beta and gamma activity. Studies completed in 1959 and 1961. Report on third study to be distributed in the near future.
Water-Surfactant	Surfactant in various waters. Study completed in 1959. Second study currently in progress.
Water Oxygen-Demand	Biochemical oxygen demand and chemical oxygen demand. Study completed in 1960.
Water Trace-Elements	Arsenic, boron, selenium, and beryllium. Study completed in 1962.
Air-Inorganics	Chloride, sulfate, fluoride, and nitrate in aqueous solution and on glass fiber hi-vol filter mats. Study completed in 1958.
Air-Lead	Filter paper tape impregnated with lead. Study completed in 1961.
Milk DDT-Residue	DDT in milk. Study completed in 1962.

PARTICIPANTS IN THIS STUDY

Air Pollution Control District, County of Los Angeles, California
American Cyanamid Company, Bound Brook, New Jersey
Arizona State Department of Health, Phoenix
Atlantic Refining Company, Philadelphia, Pennsylvania
Baltimore City Health Department
Bay Area Air Pollution Control District (San Francisco)
California State Department of Public Health (Berkeley)
California State Department of Public Health (Los Angeles)
Chicago Department of Air Pollution Control
City of Dayton, Ohio, Department of Service and Buildings
City of Philadelphia Department of Public Health
County of Fresno Department of Public Health, Fresno, California
County of Orange Air Pollution Control District, Anaheim, California
Department of Air Pollution Control, New York
El Paso City-County Health Unit Laboratory, El Paso, Texas
Erie County Laboratory, Buffalo, New York
Florida State Board of Health (Winter Haven)
Georgia Department of Public Health, Atlanta
Goodyear Atomic Corporation, Portsmouth, Ohio
Idaho Department of Health, Boise
Industrial Chemicals, Inc., South Bend, Indiana
Kennecott Copper Corporation, Salt Lake City, Utah
Maryland State Department of Health, Baltimore
Massachusetts Department of Public Health, Boston
Michigan State Department of Health, Lansing
Minnesota State Department of Health, Minneapolis
Missouri Department of Public Health and Welfare, Jefferson City
Monsanto Chemical Company, St. Louis, Missouri
New Mexico State Department of Public Health, Albuquerque
New York State Department of Health, Albany
Ohio State Department of Health, Columbus

Omaha Metropolitan Utilities District, Nebraska
Pennsylvania Department of Health, Harrisburg
Philadelphia Water Department (Torresdale Laboratory)
Research Institute for Public Health Engineering, The Hague
Richmond Field Station, University of California, Richmond
Rutgers - The State University, New Brunswick, New Jersey
Tennessee Valley Authority, Wilson Dam, Alabama (Occupational Health Branch)
Tennessee Valley Authority, Chattanooga, Tennessee (Stream Pollution Control)
Tracerlab, Inc., Waltham, Massachusetts
2794th USAF Dispensary - Class B, Kelly Air Force Base, Texas
Utah State Department of Health, Salt Lake City
Washington State Department of Health, Seattle

AIR SULFUR DIOXIDE NUMBER 1

PURPOSE OF STUDY

In the study of air pollution, sulfur dioxide is one of the major parameters for evaluating the general pollution conditions in the ambient air. Typical sources of emission of this gas to the atmosphere are the burning of fuels as in home heating plants, incinerators, power plants, automobiles and trucks, and industrial processes such as synthetic chemical plants, oil refineries, iron smelters and bleacheries.

This contaminant is a health hazard to man and to vegetation. In man, it is an irritating gas primarily affecting the upper respiratory system. In plant life, depending on concentration, it may kill all vegetation surrounding areas of high emission or in dilute concentrations may cause yellowing of leaves and retardation of plant growth.

Since sulfur dioxide is frequently used as an index of air pollution, a study to permit evaluation of precision and accuracy obtainable by analytical methods was considered desirable. A poll of Analytical Reference Service members revealed interest in a study designed to evaluate the colorimetric method of West and Gaeke.(1) An additional intent was the evaluation of the stability of the sulfur dioxide samples over a two-week period.

PREPARATION OF SAMPLE

On August 1, 1962, three concentrations of sulfur dioxide were shipped in polyethylene containers to members requesting participation. Stock solutions were prepared by dissolving a known amount of sodium bisulfite in good quality distilled water. These solutions were standardized by starch-iodine titration.

The three concentrations were made by diluting the standard stock solutions with modified 0.1M sodium tetrachloromercurate. The addition of 0.5 gm. of gum arabic to each 100 ml of 0.1M sodium tetrachloromercurate preserved the SO_2 concentrations for a period in excess of 14 days.

Due to variation of purity among different commercial sources of color-developing reagent, a sufficient quantity from one lot of 99% pure para-rosaniline hydrochloride was packed with samples to assure that this variable would

not affect participants' results. All participants were requested to report their results by August 10, 1962.

Detailed copies of the method to be evaluated were packed with each sample to assure uniform application of the specified procedure. Each participant was requested to analyze each of the three samples in triplicate and to report results to the nearest one hundredth of a microgram of SO_2 per ml of solution.

Triplicate analyses of the SO_2 samples for each concentration were performed each day during the two weeks of study by the Analytical Reference Service staff to check the stability of the samples. The analyses yielded the following results:

SO_2 CONCENTRATION, µg/ml

	Amount added: 0.25 µg/ml (Each value represents the mean of three results)	Amount added: 0.70 µg/ml (Each value represents the mean of three results)	Amount added: 1.10 µg/ml (Each value represents the mean of three results)
	A	B	C
August 2	0.29	0.81	1.15
August 3	0.31	0.81	1.15
August 6	0.29	0.80	1.15
August 7	0.26	0.75	1.07
August 8	0.28	0.75	1.09
August 9	0.22	0.64	0.97
August 10	0.30	0.78	1.10
August 13	0.31	0.78	1.16
August 14	0.28	0.77	1.10
August 15	0.27	0.74	1.09
August 16	0.28	0.73	1.06
August 17	0.29	0.75	1.10
Mean	0.28	0.76	1.10

TREATMENT OF DATA

The statistical terms, as used in this report, are defined in the Glossary (page 31). The method for rejection of outliers, which seems to warrant more detailed treatment, is discussed below.

Rejection of Outliers

In statistical analyses of the data developed by the Analytical Reference Service in various studies, the results of a few laboratories are often noted to be very erratic for no discernible reason. Examination of many references indicated that no absolute criterion exists by which erratic values (outliers) can be discarded and, further, that statisticians disagree as to whether a suspect value can be discarded. But even one very erratic result can give a false and unfair picture of a method. Since evaluation of methods is the purpose of the Analytical Reference Service, it has been decided to use what seems to be a fair, workable, and statistically sound method for the elimination of outliers. Such a method involves use of a probability curve and judgment based on knowledge of the sensitivity of the method and of the physical factors which might affect the sample.

The Dixon Q test for the rejection of outliers is not used because tables are not available for the high number of replicates included in ARS studies.

Ideally, the plot of the frequency of measurements against their magnitudes gives the bell-shaped normal curve. The validity of many statistical procedures is based on this normal distribution curve, although it has been shown that moderate departures from normality do not seriously affect validity. If, on probability paper, the results of an experiment are plotted against the corresponding plotting interval on a frequency scale, a normal distribution curve approximates a straight line. Just as an erratic result is far removed from the others on the normal curve, so is it also far removed from the others on the straight-line curve. A judgment to accept or reject a result is based on this graphical picture and a knowledge of the sensitivity and limitations of the method.

DISCUSSION OF RESULTS

Nineteen of the forty-three agencies participating in this study reported that they routinely analyze for sulfur dioxide, an indication that approximately 50% of the participants perform this determination only occasionally and approximately 50% were performing it for the first time. Sixteen of the nineteen reported the West-Gaeke Method[1] as the one in use in their laboratories.

One of the goals initially established by the Analytical Reference Service and by the early members for satisfactory laboratory performance was the achievement of values within the best 50% of reported data. These values are noted under the discussion of each of the three samples.

SAMPLE A (0.25 μg/ml SO_2 added)

It was necessary to reject three sets of data by the statistical method discussed under Statistical Treatment of Data, Rejection of Outliers (page 3). Two of the laboratories reporting the rejected data do not routinely analyze for SO_2 and the third uses a different analytical method. The three rejected means are circled on the attached Probability Curve. They appear near the bottom of the left portion of the curve.

The statistical values for all acceptable reported results are summarized below:

SAMPLE A

Number of participants included in statistical analysis	=	40
Number of results	=	123
Mean	=	0.30 μg/ml
Median	=	0.29 μg/ml
Range	=	0.27 μg/ml
Standard deviation	=	0.053 μg/ml
Coefficient of variation	=	17.6%
Average percent deviation from amount added	=	20.8%
95% Confidence Interval	=	[0.291 - 0.309 μg/ml]

The replicate results reported by each participant for Sample A are tabulated in Table 1. At this low concentration, the graph plotted from the amount added reveals a distribution skewed in the direction of the higher values. It should be noted, however, that the determined mean varies from the amount added by only $\pm$ 0.05 µg/ml. Fifty percent of the participants reported values within $\pm$ 0.04 µg/ml of concentration added.

Table 1. SAMPLE A (Concentration SO_2 added = 0.25 μg/ml)

Lab No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
9523	0.29 0.30 0.28	0.29	2	16.0
3511	0.31 0.35 0.31	0.32	5	29.2
3611	0.26 0.27 0.26	0.26	1.1	5.2
1426	0.36 0.36 0.36	0.36	0	44.0
09	0.27 0.27 0.28	0.27	1.1	9.2
4626	0.24 0.26 0.24	0.25	4	4.0
4311	0.30 0.30 0.29	0.30	1.0	18.4
06	0.28 0.29 0.30	0.29	2	16.0

Table 1 (Cont'd). SAMPLE A (Concentration SO_2 added = 0.25 μg/ml)

Lab No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
	0.34			
03	0.30	0.34	6.7	34.4
	0.37			
	0.30			
6911	0.30	0.30	0	20.0
	0.30			
	0.30			
4116	0.30	0.30	0	20.0

	0.27			
2526	0.24	0.26	5	6.4
	0.27			
	0.32			
1211	0.32	0.32	0	28.0
	0.32			
	0.32			
4611	0.33	0.32	1.8	28.0
	0.31			
	0.40			
6311	0.37	0.39	3.3	56.0
	0.40			
	0.41			
3926	0.40	0.41	2.4	65.2
	0.43			

Table 1 (Cont'd). SAMPLE A (Concentration SO_2 added = 0.25 μg/ml)

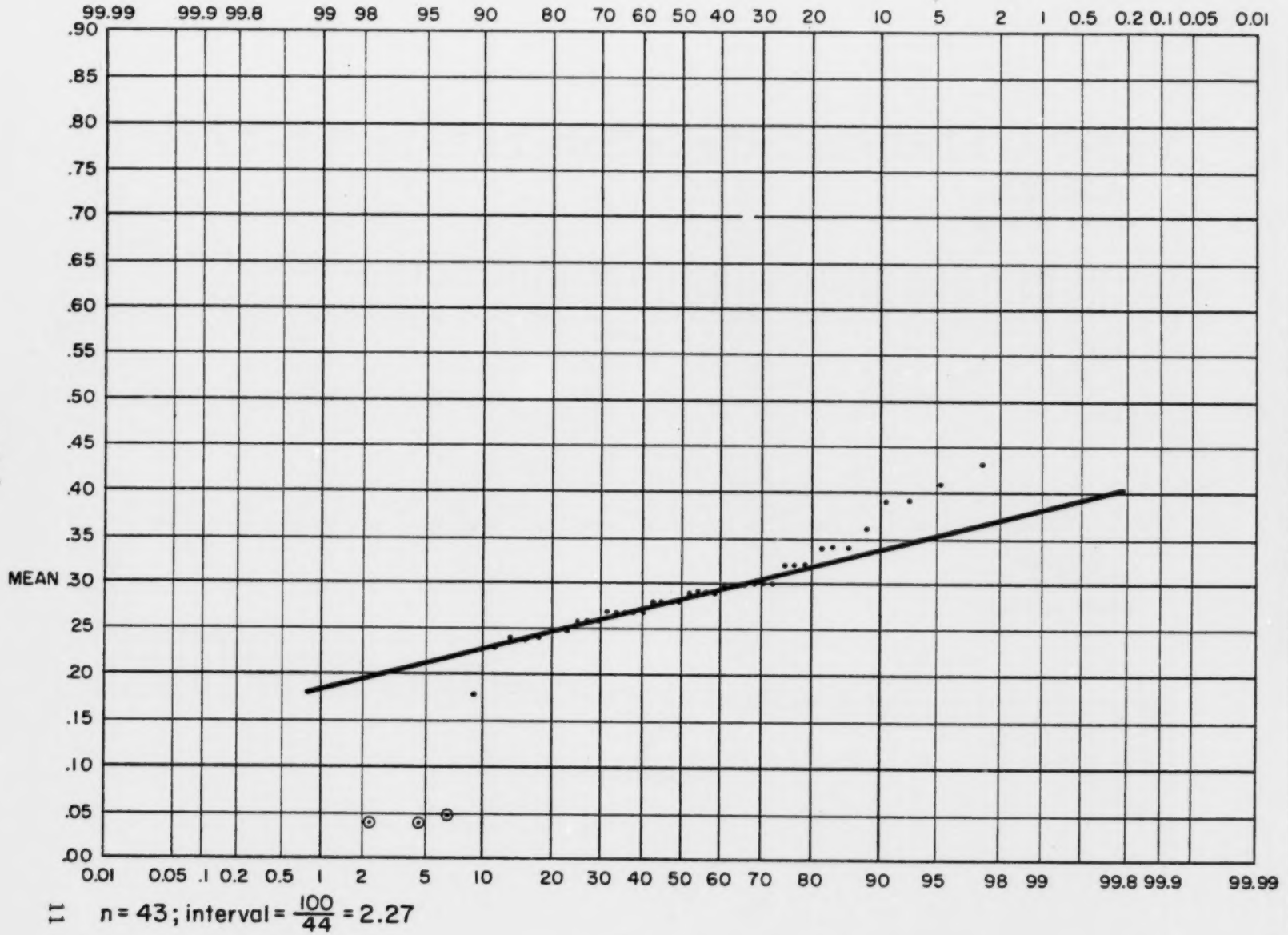
Lab No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
	0.43			
2626	0.43	0.43	.6	73.2
	0.44			
	0.29			
8512	0.27	0.28	2.1	12.0
	0.28			
	0.23			
8313	0.23	0.24	6.6	8.0
	0.27			
	0.30			
7142	0.30	0.30	0	20.0
	0.30			
	0.26			
7124	0.27	0.27	2.2	8.0
	0.28			
	0.25			
5111	0.24	0.24	1.2	2.4
	0.24			
	0.34			
05	0.34	0.34	0	36.0
	0.34			
	0.25			
7122	0.28	0.28	5.7	10.4
	0.30			

Table 1 (Cont'd). SAMPLE A (Concentration SO_2 added = 0.25 μg/ml)

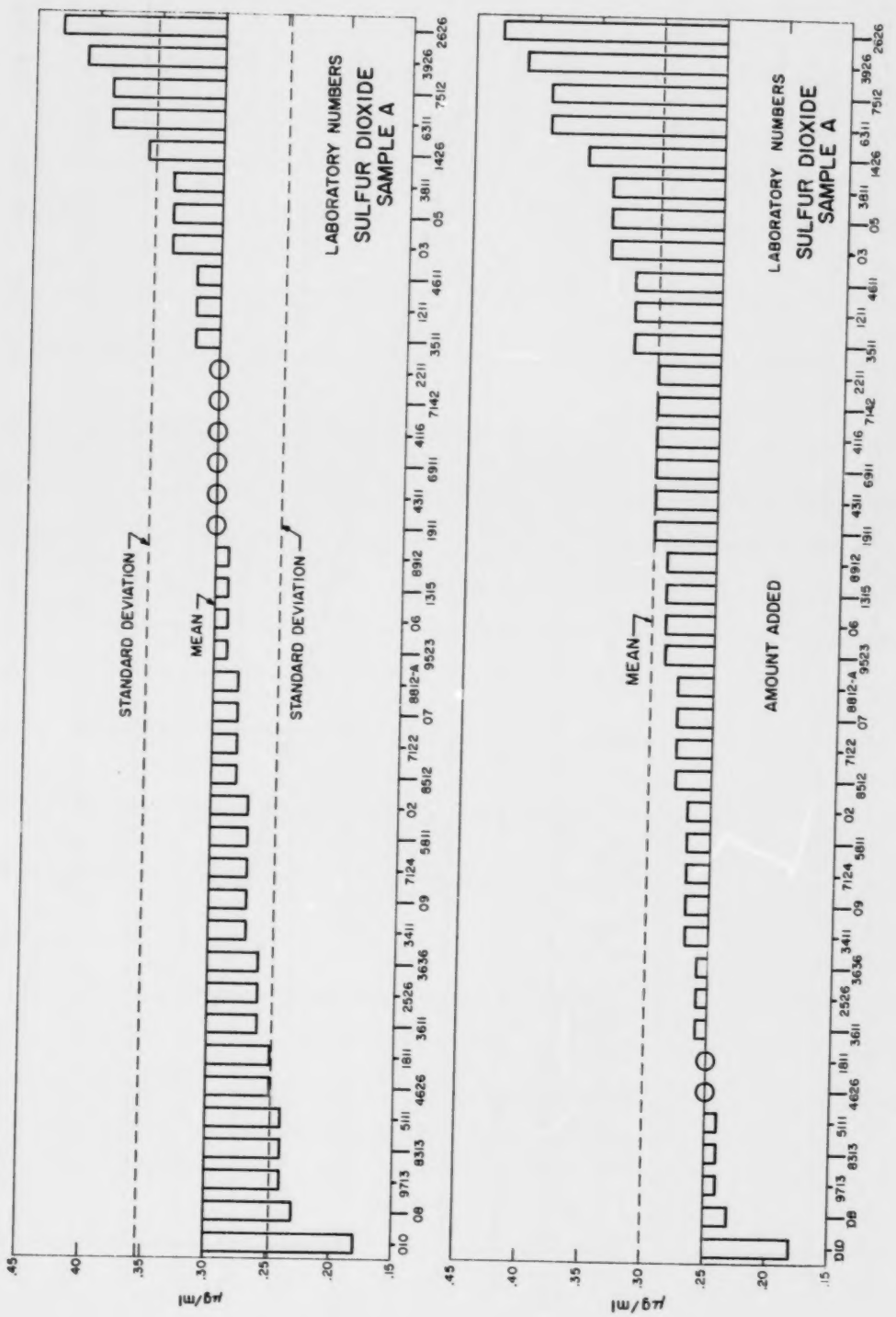
Lab No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
7512	0.40 0.40 0.30 0.45	0.39	14.3	54.8
1811	0.24 0.25 0.26	0.25	2.4	2.4
1315	0.29 0.30 0.29	0.29	1	17.2
3636	0.26 0.26 0.26	0.26	0	4.0
3811	0.33 0.35 0.34	0.34	1.7	36.0
07	0.27 0.28 0.28	0.28	1	10.4
8912	0.30 0.29 0.29	0.29	1	17.2
2211	0.31 0.30 0.29	0.30	2	20.0

Table 1 (Cont'd). SAMPLE A (Concentration SO_2 added = 0.25 μg/ml)

Lab No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
03	0.23 0.23 0.24	0.23	1.3	6.4
3411	0.27 0.27 0.26	0.27	1.1	6.4
1911	0.30 0.30 0.30	0.30	0	20.0
9713	0.24 0.24 0.24	0.24	0	4.0
010	0.18 0.18 0.18	0.18	0	28.0
5811	0.28 0.28 0.24	0.27	5.9	9.2
8812 (A)	0.22 0.28 0.30 0.31 0.28 0.29	0.28	7.1	16.0
02	0.28 0.26 0.26	0.27	3.7	6.4



PROBABILITY CURVE
SAMPLE A


STANDARD DEVIATION
MEAN
STANDARD DEVIATION
LABORATORY NUMBERS
SULFUR DIOXIDE
SAMPLE A
μg/ml
MEAN
AMOUNT ADDED
LABORATORY NUMBERS
SULFUR DIOXIDE
SAMPLE A
μg/ml

SAMPLE B (0.70 μg/ml SO_2 added)

The three means rejected for Sample B were reported by the same three laboratories as for Sample A. These three values were extremely low, as shown on the attached Probability Curve. Inclusion of these results would have introduced a substantial degree of bias in the evaluation of the method:

The statistical data derived for this sample are given below:

Number of participants	=	40
Number of results	=	121
Mean	=	0.74 μg/ml
Median	=	0.72 μg/ml
Range	=	0.58 μg/ml
Standard deviation	=	0.117 μg/ml
Coefficient of variation	=	15.8%
Average % deviation from amount added	=	12.1%
95% Confidence Interval	=	[0.719 - 0.761 μg/ml]

An approximately normal distribution of results is indicated by the closeness of the mean and median, 0.74 and 0.72 μg/ml respectively. Good precision was evidenced by the standard deviation and by the position of the true mean between 0.719 and 0.761 μg/ml 95% of the time.

Accuracy was excellent, with the mean for the sample only 0.04 μg/ml greater than the concentration added (0.70 μg/ml).

Individual results reported for this sample are tabulated in Table 2. Bar graphs for this sample are plotted from the mean and the amount added. Fifty percent of the laboratories reported concentrations of SO_2 within ± 0.05 μg/ml of the amount added.

Table 2. SAMPLE B (Concentration SO_2 added = .70 μg/ml)

Lab. No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
9523	0.77 0.75 0.76	0.76	.7	8.5
3511	0.74 0.79 0.74	0.76	3.0	8
3611	0.65 0.66 0.64	0.65	.9	7.1
1426	0.74 0.79 0.72	0.75	3.4	7.1
09	0.71 0.71 0.72	0.71	.4	1.8
4626	0.64 0.62 0.62	0.63	1.5	10.4
4311	0.74 0.72 0.73	0.73	.8	4.2
06	0.76 0.78 0.76	0.77	1.2	9.4

Table 2 (Cont'd). SAMPLE B (Concentration SO_2 added = .70 μg/ml)

Lab. No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
	0.89			
03	0.84	0.87	1.8	23.7
	0.87			
	0.71			
6911	0.70	0.70	.4	.4
	0.70			
	0.75			
4116	0.78	0.77	1.6	10
	0.78			
	0.72			
2526	0.72	0.73	1.3	3.7
	0.74			
	0.76			
1211	0.78	0.77	1.2	9.4
	0.76			
	0.76			
4611	0.77	0.76	.3	9.0
	0.76			
	0.90			
6311	0.90	0.90	0	28.5
	0.90			
	0.94			
3926	1.00	0.97	2.0	38.5
	0.97			

Table 2 (Cont'd). SAMPLE B (Concentration SO_2 added = .70 μg/ml)

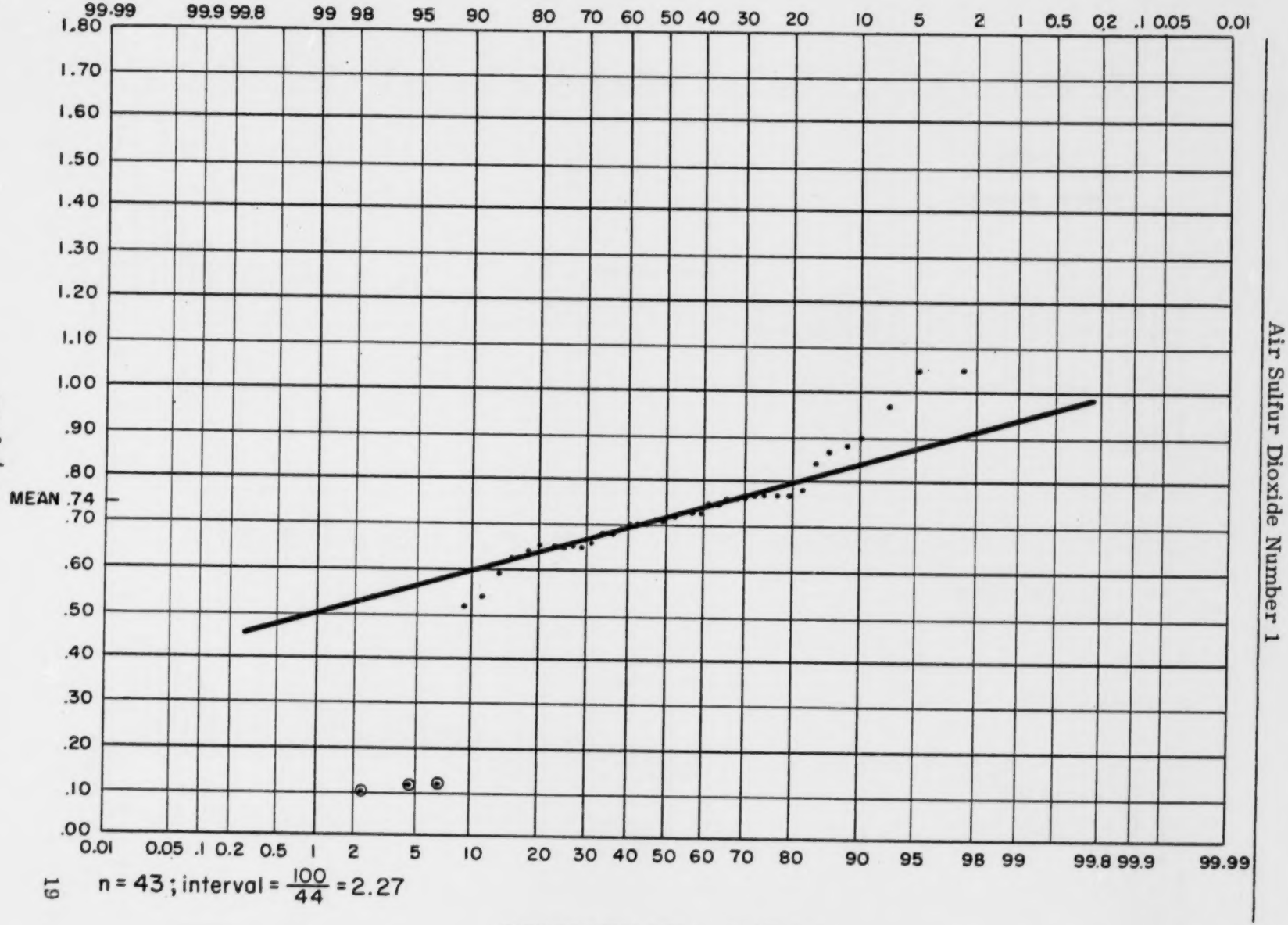
Lab. No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
2626	1.04 1.06 1.06	1.05	.9	50.4
8512	0.73 0.71 0.71	0.72	1.3	2.2
8313	0.68 0.65 0.63	0.65	2.4	6.5
7142	0.72 0.73 0.75	0.73	1.3	4.7
7124	0.64 0.68 0.66	0.66	1.9	5.7
5111	0.67 0.66 0.70	0.68	1.9	3.2
05	1.08 1.04 1.04	1.05	1.5	50.4
7122	0.74 0.76 0.74	0.75	1.3	6.5

Table 2 (Cont'd). SAMPLE B (Concentration SO_2 added = .70 μg/ml)

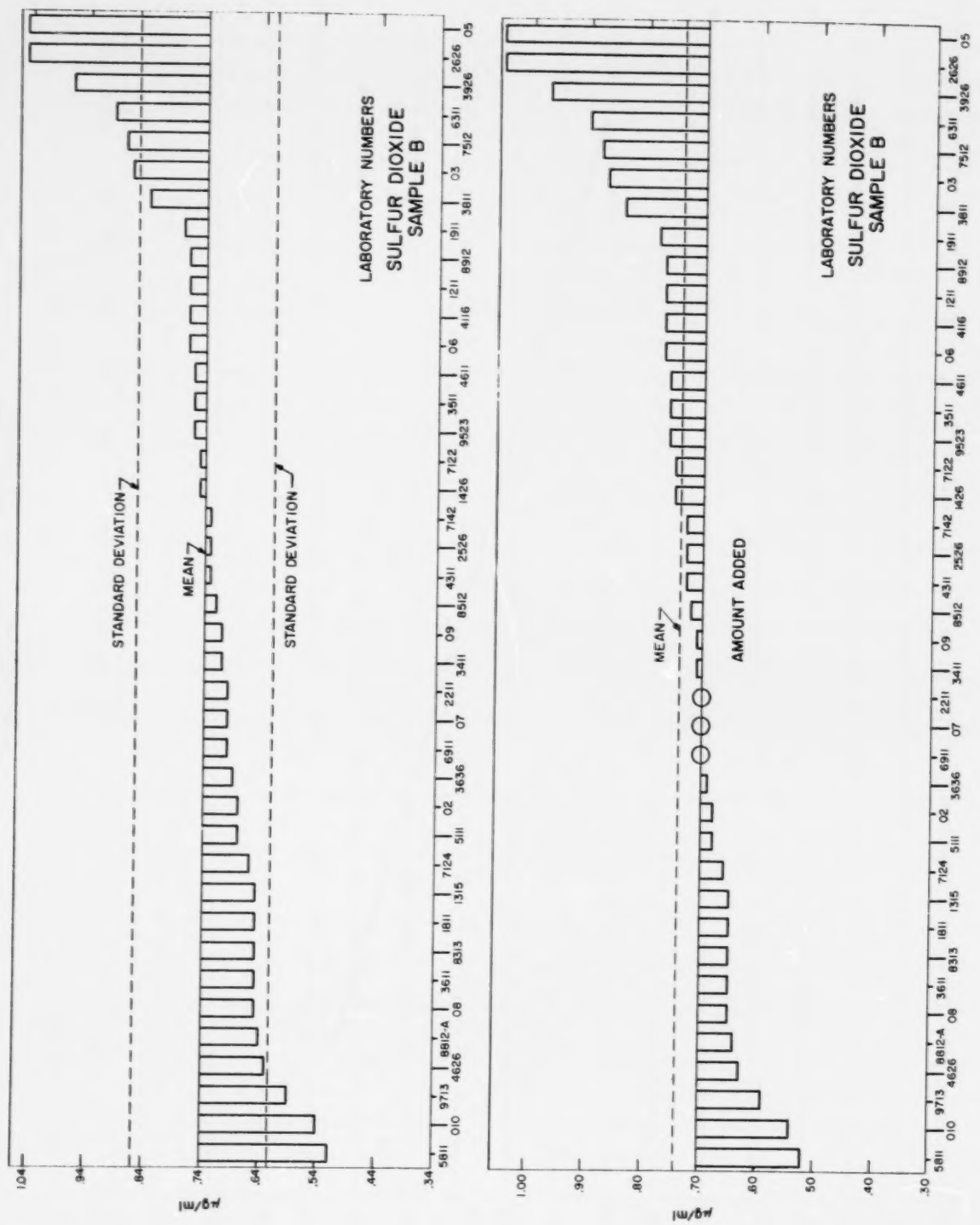
Lab. No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
	0.90			
7512	0.80	0.88	11.3	33.2
	1.00			
	0.80			
	0.65			
1811	0.64	0.65	.9	7.1
	0.66			
	0.67			
1315	0.63	0.65	2.4	7.5
	0.64			
	0.68			
3636	0.70	0.69	1.4	1.8
	0.68			
	0.85			
3811	0.86	0.84	2.3	20
	0.81			
	0.70			
07	0.70	0.70	0	0
	0.70			
	0.77			
8912	0.77	0.77	.9	10
	0.77			
	0.71			
2211	0.71	0.70	2.2	2.2
	0.67			

Table 2 (Cont'd). SAMPLE B (Concentration SO_2 added = .70 μg/ml)

Lab. No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
08	0.63 0.66 0.66	0.65	2	7.1
3411	0.71 0.73 0.69	0.71	1.8	2.2
1911	0.76 0.80 0.78	0.78	1.6	11.4
9713	0.58 0.59 0.60	0.87	1.0	15.7
010	0.54 0.54 0.54	0.54	0	22.8
5811	0.50 0.50 0.56	0.52	5	25.7
8812(A)	0.64 0.64 0.65	0.64	.4	8
02	0.67 0.69 0.69	0.68	1.4	2.2



PROBABILITY CURVE
SAMPLE B


STANDARD DEVIATION
MEAN
STANDARD DEVIATION
LABORATORY NUMBERS
SULFUR DIOXIDE
SAMPLE B
MEAN
AMOUNT ADDED
LABORATORY NUMBERS
SULFUR DIOXIDE
SAMPLE B

SAMPLE C (1.10 μg/ml SO_2 added)

The outliers that were rejected for Sample C increased from three to five. Data from the same three laboratories (noted under samples A and B) were rejected due to their extremely low results, and two laboratories reported mean values which were so erratically high as to necessitate rejection. These five means are circled on the Probability Curve.

The accuracy and precision obtained at this concentration were excellent. The mean varied from the amount added by only 0.01 μg/ml. The average percent deviation from the amount added was 10.4%.

As the mean and median were very close, 1.09 and 1.07 μg/ml respectively, approximately equal numbers of results appear above and below the mean, indicating a balanced distribution. Statistical data for this sample are summarized below:

Number of participants	=	38
Number of results	=	115
Mean	=	1.09 μg/ml
Median	=	1.07 μg/ml
Range	=	0.77 μg/ml
Standard deviation	=	0.140 μg/ml
Coefficient of variation	=	12.8% μg/ml
Average percent deviation	=	10.4%
95% Confidence interval	=	[1.064 - 1.116 μg/ml]

Table 3 details the individual results reported by participants for this sample. The bar graph drawn from the mean illustrates precision; the one drawn from the amount added shows accuracy. At this concentration, 50% of the participants reported mean values of $\pm$ 0.07 µg/ml.

Table 3. SAMPLE C (Concentration SO_2 added = 1.10 μg/ml)

Lab. No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
9523	1.11 1.14 1.15	1.13	1.4	3.0
3511	1.12 1.20 1.10	1.14	3.5	3.6
3611	0.96 0.93 0.89	0.93	2.4	15.7
1426	1.15 1.13 1.09	1.12	2.0	2.7
09	1.02 1.02 1.05	1.03	1.2	6.3
4626	0.92 0.93 0.94	0.93	.6	15.4
4311	1.05 1.03 1.04	1.04	.5	5.4
06	1.05 1.03 1.05	1.04	.9	5
03	1.10 1.17 1.15	1.14	2.2	3.6

Table 3 (Cont'd). SAMPLE C (Concentration SO_2 added = 1.10 μg/ml)

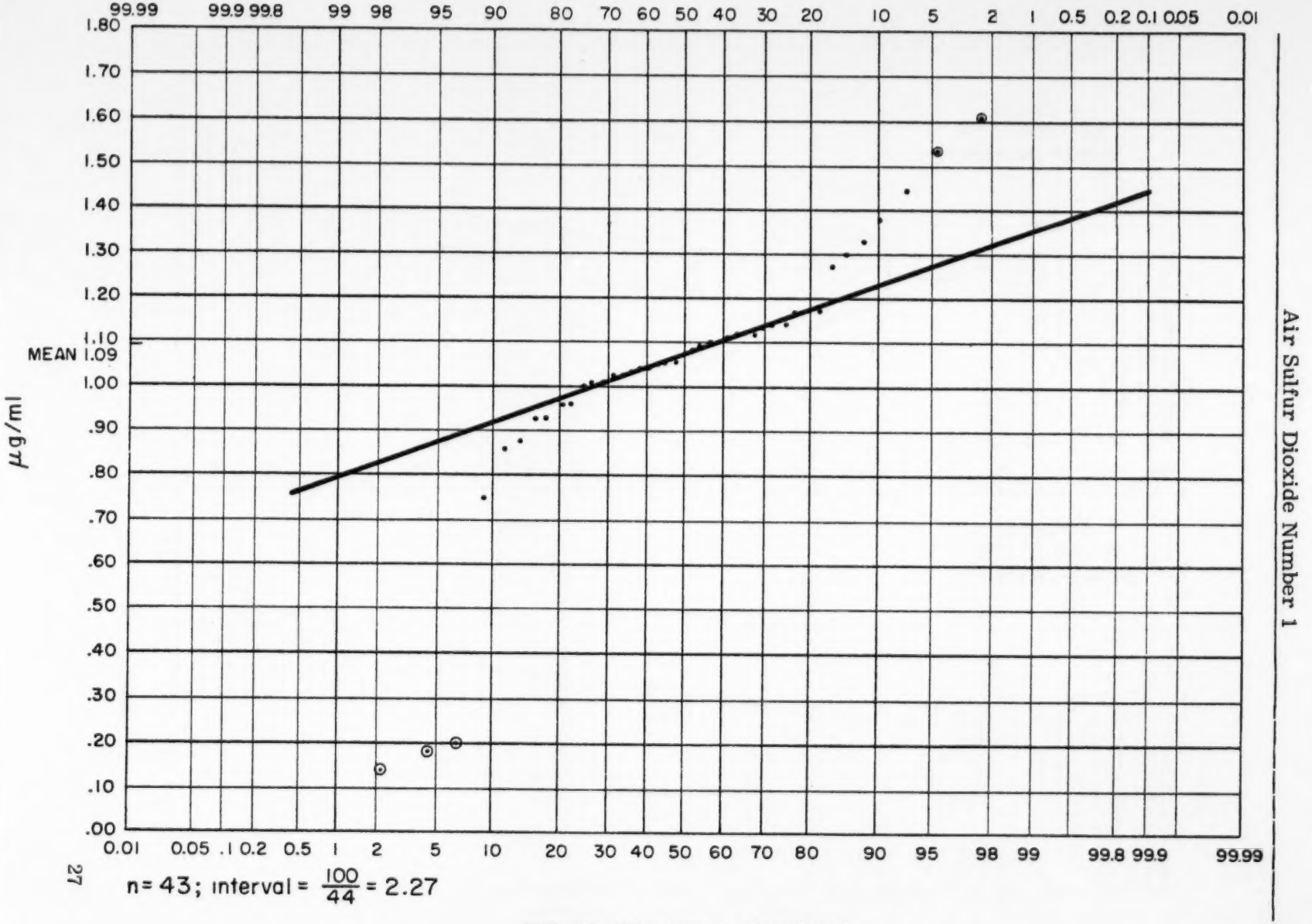
Lab. No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
6911	1.05 1.06 1.04	1.05	.5	4.5
4116	1.20 1.20 1.12	1.17	3	6.6
2526	1.09 1.05 1.05	1.06	1.5	3.2
1211	1.12 1.13 1.12	1.12	.2	2.0
4611	1.11 1.13 1.11	1.12	.8	1.4
6311	1.34 1.34 1.30	1.33	1.2	20.5
3926	1.44 1.41 1.47	1.44	1.3	30.9
8512	1.05 1.04 1.06	1.05	.5	4.5
8313	1.04 1.01 0.99	1.01	1.5	7.8

Table 3 (Cont'd). SAMPLE C (Concentration SO_2 added = 1.10 μg/ml)

Lab. No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
	1.11			
7142	1.11	1.11	0	0.9
	1.11			
	1.01			
7124	1.01	1.00	1	8.7
	0.99			
	1.11			
5111	1.09	1.08	2.1	2.0
	1.05			
	1.15			
7122	1.18	1.17	1.1	6.3
	1.18			
	1.40			
7512	1.30	1.38	3.3	33.2
	1.40			
	1.40			
	0.96			
1811	0.96	0.96	.3	12.3
	0.97			
	0.99			
1315	0.95	0.96	1.6	12.3
	0.95			
	1.30			
3636	1.33	1.30	1.2	18.4
	1.28			
	1.31			
3811	1.25	1.27	1.8	15.7
	1.26			
	1.09			
07	1.10	1.10	.05	0.5
	1.11			

Table 3 (Cont'd). SAMPLE C (Concentration SO_2 added = 1.10 μg/ml)

Lab. No.	μg SO_2/ml	Mean	Percent deviation within lab. (precision)	Percent deviation from amount added (accuracy)
8912	1.11 1.05 1.11	1.09	2.3	2.0
2211	1.10 1.10 1.10	1.10	0	0
08	1.00 1.02 1.03	1.02	.9	7.5
3411	0.98 1.04 1.05	1.02	2.9	6.9
1911	1.15 1.17 1.19	1.17	1.1	6.3
9713	0.87 0.87 0.87	0.87	1.1	20.2
010	0.86 0.86 0.86	0.86	0	21.8
5811	0.78 0.78 0.70	0.75	4.8	31.4
8812 (a)	1.05 1.06 1.07	1.06	.5	3.6
02	1.00 1.00 1.04	1.01	1.5	7.8



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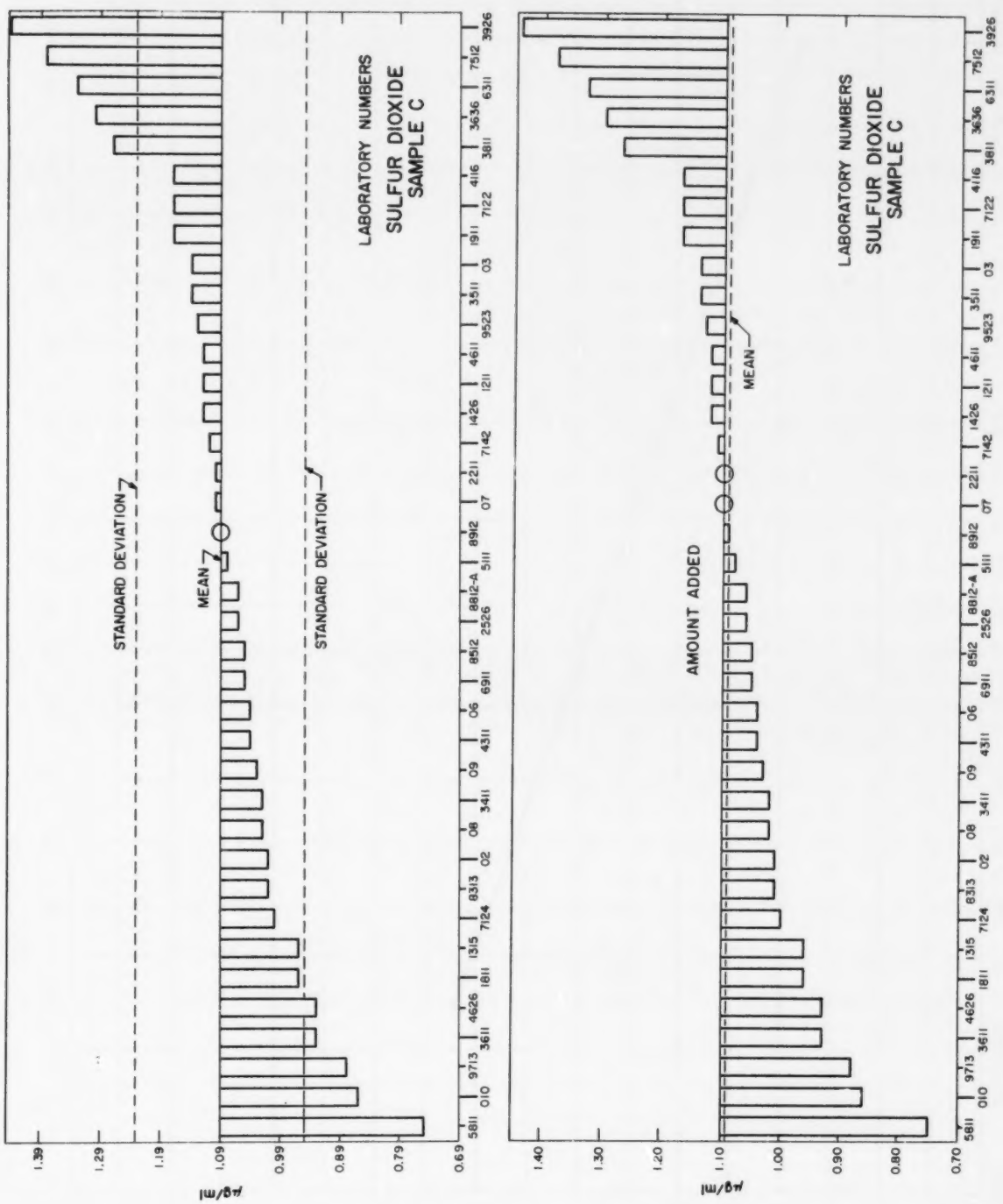
PROBABILITY CURVE
SAMPLE C


STANDARD DEVIATION
MEAN
STANDARD DEVIATION
LABORATORY NUMBERS
SULFUR DIOXIDE
SAMPLE C
μg/ml
1.39
1.29
1.19
1.09
0.99
0.89
0.79
0.69
AMOUNT ADDED
MEAN
LABORATORY NUMBERS
SULFUR DIOXIDE
SAMPLE C
μg/ml
1.40
1.30
1.20
1.10
1.00
0.90
0.80
0.70

SUMMARY

The SO_2 sample was prepared without the addition of nitrogen dioxide, the one significant interference for the West-Gaeke[1] method. West and Ordoveza[2] report a modified procedure for the elimination of this interference by the addition of sulfamic acid to decompose nitrite.

Perry and Tabor[3] found that about 15% of the sulfur dioxide content is lost from samples after two weeks of storage. To overcome this loss, the study attempted to stabilize the sample by adding 0.5 gm. of gum arabic[4] per 100 ml of 0.1 M sodium tetrachloromercurate as a preservative. Addition of this preservative was apparently effective as the analyses of the three concentrations by the ARS staff failed to show any loss of SO_2 after 16 days storage.

The standard deviation increased through the three samples as the concentration increased. Precision as indicated by the coefficient of variation improved with each increase of concentration.

Data tabulated in Table 4 indicate that this method is accurate and precise even when used by laboratories not routinely determining SO_2 or performing the determination for the first time.

In their comments the cooperating agencies stressed the simplicity, sensitivity, and reproducibility of the method. They indicated that their greatest difficulty arose in the standardization of the stock sodium bisulfite solution.

Table 4. Summary of Statistical Data

Air, SO_2 Number 1

Sample Concentration	No. of Participants Included in Statistical Analysis	Mean	Median	Range	Standard Deviation	Coefficient of Variation	Average % Deviation	95% Confidence Interval
A (0.25 μg/ml)	40	0.30	0.29	0.27	0.053	17.6%	13.0%	[0.291 - 0.309 μg/ml]
B (0.70 μg/ml)	40	0.74	0.72	0.58	0.117	15.8%	12.1%	[0.719 - 0.761 μg/ml]
C (1.10 μg/ml)	38	1.09	1.07	0.77	0.140	12.8%	10.4%	[1.064 - 1.116 μg/ml]

GLOSSARY OF STATISTICAL TERMS

For the convenience of our members, the Analytical Reference Service has compiled a glossary of statistical terms with the definition of their meaning as used in our reports, to insure more uniform and complete understanding.

ACCURACY Accuracy is the correctness of a measurement, or the degree of correspondence between the result and the true value.

ACCURACY DATA*

Measurements which relate to the difference between the average test results and the true result when the latter is known or assumed. The following measures apply:

Mean error* - The average difference with regard to sign of the test results from the true results. Also equal to the difference between the average of a series of test results and the true result.

Relative error* - The mean error of a series of test results as a percentage of the true result.

PRECISION Precision is a measure of the reproducibility of measurements, or the degree to which the measurements correspond to one another.

PRECISION DATA*

Measurements which relate to the variation among the test results themselves, i.e., the scatter or dispersion of a series of test results, without assumption of any prior information. The following measures apply:

Variance* - The sum of squares of deviations of the test results from the mean of the series, after divisions by one less than the total number of test results.

Standard deviation* - The square root of the variance.

Coefficient of variation* - The standard deviation of a series of test results as a percentage of the mean of this series.

Range* - The difference in magnitude between the highest test result and the lowest test result in a series.

Mean* - The sum of a series of test results divided by the number in the series. Arithmetic mean is understood.

Median - The value above and below which an equal number of observations lie.

Percent deviation within laboratory - The average difference between a laboratory's replicate results and the mean, expressed as a percentage of the mean.

Confidence limits - Limits within which the true mean of the series will lie with a given probability.

*Annual Reviews, Analytical Chemistry, April 1962. pg. 364R.

REFERENCES

1. Annual Reviews. Analytical Chemistry. pg. 364R. April 1962.

2. Bennett, C.A., and Franklin, N.L. Statistical Analysis in Chemistry and the Chemical Industry. John Wiley & Sons, Inc. New York. 1954.

3. Bauer, E.L. A Statistical Manual for Chemists. Academic Press. New York. 1960.

4. Dixon, W.J., and Massey, F.J. Introduction to Statistical Analysis. McGraw-Hill Book Co., Inc. New York. 1951.

COMMENTS OF PARTICIPANTS

LABORATORIES

No. 1211

The preparation of the dye gives a product superior to that obtained by our method. Bi-iodate standardization of the SO_2 appears to be more satisfactory than direct iodine titration. Because of the heat produced using concentrated H_2SO_4, an equivalent amount of 1:1 H_2SO_4 is suggested.

No. 1426

No apparent difficulty was encountered using this procedure. In making up the standard SO_2 solution, the sodium (meta) bisulfite was added to water which had first been de-aerated by sweeping with nitrogen.

No. 1811

The method as presented requires that concentrations of the potassium bi-iodate and of the sodium thiosulfate be identical. If this is not the case, the bi-iodate titer of the thiosulfate must be determined in order to determine the amount of bi-iodate consumed. Then the first line under "calculations" should be modified as follows:

$$V_1N_1 - V_2N_2 = V_3N_3$$

No. 1911

Simple and straightforward.

No. 2311

Timing should be stressed, it still is not stable in 25 minutes, but if read in exactly 25 minutes, duplication is good.

No. 2626

In the standardization procedure for the sulfur dioxide solution (prepared from $Na_2S_2O_5$), excessively large titers resulted from the use of 0.025N sodium thiosulfate and reproducibility was extremely difficult. This difficulty was surmounted by using 0.1N sodium thiosulfate.

No. 3411

Samples contained slight precipitate when analysis was begun. Use of .025N potassium bi-iodate is questioned. We used a standard solution of potassium iodate instead.

No. 3511

This was our first experience with this method. The method seemed to be extremely sensitive. This may have been due to the fact that we were using a Beckman D.U. with a photomultiplier attachment. However, we had very little trouble with the method except several attempts were made before we could establish a satisfactory standard curve. One thing we did find was that the stock $NaHSO_3$ Solution is unstable. It is necessary to restandardize each time just before making the dilutions.

No. 3636

When developed samples are left in the spectrophotometer for 5-10 minutes a decrease in Optical Density is often noted. However, when fresh aliquots from the same developed solution are re-measured, the original value is observed.

No. 3811

Unable to obtain potassium bi-iodate solution. Therefore, standardized the sodium (meta) bisulfate solution against iodine (0.01N) with respect to SO_2.

No. 4116

Due to the unavailability of potassium bi-iodate, the sodium bisulfate could not be standardized. Nitrogen was bubbled through the distilled water that was used to make up the standard solutions.

No. 4311

Samples showed some turbidity. This cleared during the reaction period except for Sample "A". Method seems simple and workable.

No. 4611

(1) Twelve hours is inconvenient for having dye stand before filtration; either 24 hours or within a normal working day is preferable.

(2) Regarding pipetting 10 μg SO_2/ml standard solution - can use burette for safety and accuracy.

No. 4626

Different bottles of p-rosaniline hydrochloride give different standard curves.

No. 5111

The method is very simple and easy to do after once establishing a good curve. Since there seems to be few interferences, it should make a reliable test for routine work.

We found it necessary to centrifuge the samples before reading them on the spectrophotometer. A fine precipitate formed during the 25 minute time for color development.

No. 5811

We have found this method to be a reliable and relatively rapid procedure. It has been used in our laboratory on a routine basis.

No. 6311

For analysis, 1-inch cuvettes with a Bausch & Lomb Spectronic "20" were utilized in lieu of 1 cm. cells.

No. 7124

Our investigation of method showed maximum color development occurred at 20 minutes.

No. 7142

This method is about the fastest for the very small SO_2 Concentrations we are concerned with, and still gives reasonable accuracy.

In your standardization, however, the method could be improved by use of large volumetric glassware as it is rather difficult to measure out accurately the very small volumes used.

No. 7512

The standard curve was non-linear, especially at the highest concentration.

No. 8313

This is a rapid and simple method for the determination of low concentrations of SO_2. Some fading of the color was noted for periods greater than 30 minutes after the development of the color.

No. 8512

Results seem to be reproducible, and the method quite satisfactory.

No. 8912

Spectrophotometer not available. Used Lumetron Model 15 Colorimeter with fixed filter (480 to 520 mμ).

No. 02

Samples A, B, and C each contained a flocculent precipitate which caused turbidity in the solutions. The results reported are not corrected for this turbidity.

No. 03

We obtained better color development with the supplied p-rosaniline than in any previous determination.

No. 06

Method is straightforward, simple and very satisfactory.

DETERMINATION OF SULFUR DIOXIDE BY THE WEST-GAEKE METHOD[1]

(Colorimetric Determination)

I. REAGENTS

A. Sodium Tetrachloromercurate (0.1 M)

Dissolve 27.2 g (0.1 mole) mercuric chloride and 11.7 g (0.2 mole) sodium chloride in a one-liter volumetric flask and dilute to the mark with distilled water.

B. Hydrochloric Acid Bleached p-Rosaniline

Dissolve 0.25 g of para-rosaniline hydrochloride in 100 ml of distilled water, and let stand for 12 hours. Filter through filter paper. Pipet 4 ml of the filtrate into a 100 ml volumetric flask. Add 6 ml of concentrated hydrochloric acid. Allow to stand 5 minutes then dilute to the mark with distilled water.

C. Formaldehyde (0.2%)

Pipet 5 ml of 40% formaldehyde into a one-liter volumetric flask and dilute to the mark with distilled water

D. Sodium Bisulfite ($NaHSO_3$) or Sodium (meta) Bisulfite ($Na_2S_2O_5$), Reagent Grade.

E. 0.025 N Potassium Bi-iodate Solution

F. Concentrated Sulfuric Acid

G. Potassium Iodide

H. 0.025 N Sodium Thiosulfate Solution

1. Starch indicator

II. EQUIPMENT

A. Spectrophotometer

B. 5-25 ml Volumetric Flask

C. 5-25 or 50 ml Erlenmeyer Flask

D. A Set of Matched 1 cm Cells for Spectrophotometer

III. PROCEDURE

A. Standard Sulfur Dioxide Solution

Dissolve approximately 0.160 g of sodium bisulfite ($NaHSO_3$) or 0.149 g of sodium (meta) bisulfite ($Na_2S_2O_5$) in one liter of distilled water. This solution is approximately 100 μg/ml with respect to SO_2, but must be standardized as dissolved oxygen in the water oxidizes some $SO_3^=$ to $SO_4^=$.

B. Standardization of Sulfur Dioxide Solution

1. Place a 50 ml aliquot of approximately 100 μg/ml SO_2 solution in an Erlenmeyer flask.

2. Add 10 ml of 0.025 N potassium bi-iodate solution.

3. Add 2 ml of concentrated sulfuric acid.

4. Add approximately 0.1 g of potassium iodide crystals.

5. If no orange-yellow color appears, add another 10 ml aliquot of bi-iodate.

6. To this solution, add from a buret, 0.025 N sodium thiosulfate solution until the solution becomes pale yellow

7. Add about one ml of starch indicator; the solution should turn blue.

8. If the solution does not turn blue, the end point has been overrun and the standardization must be begun again.

9. Continue adding sodium thiosulfate until the blue color disappears. Record volume of thiosulfate used.

10. Calculations

$$V_1 - V_2 = V_3$$

$$V_3 \times N_3 = V_{SO_2} \times N_{SO_2}$$

$$N_{SO_2} \text{ (equiv/l)} \times \frac{64.07}{2} \text{ (g/equiv)} = \text{g/l}$$

$$\text{grams/L} \times 10^3 = \mu\text{g/ml}$$

V_1 = ml of potassium bi-iodate added

V_2 = ml of sodium thiosulfate used

V_3 = ml of potassium bi-iodate consumed

N_3 = normality of bi-iodate solution (0.025 N)

V_{SO_2} = ml of SO_2 solution added

N_{SO_2} = normality SO_2 solution

C. The Standard Curve

1. Prepare by dilution with 0.1 M sodium tetrachloromercurate, a 10 μg SO_2/ml solution from the SO_2 solution which has been standardized. For example: if the SO_2 solution which was

standardized was 100 μg SO_2/ml: to make a 10 μg SO_2/ml solution pipet 10 ml of 100 μg SO_2/ml solution into a 100 ml volumetric flask and dilute to the mark with 0.1 M sodium tetrachloromercurate.

2. Into a series of 25 ml volumetric flasks, pipet successively the following volumes (ml) of the 10 μg SO_2/ml standard solution: 0.0, 0.25, 0.75, 2.0 and 3.0.

3. Dilute all of the 25 ml flasks to the mark with 0.1 M sodium tetrachloromercurate. The concentration, μg of SO_2/ml in each of these volumetric flasks is: 0, 0.10, 0.30, 0.80, and 1.2, respectively.

4. Pipet 10 ml of known SO_2 solution from each 25 ml volumetric flask into separate 25 or 50 ml Erlenmeyer flasks (Label correctly).

5. To each Erlenmeyer flask, pipet in the following order: (a) 1.0 ml of bleached para-rosaniline solution, (b) 1.0 ml of 0.2% formaldehyde solution.

6. Allow 25 minutes for color development.

7. Determine optical density of standards on a spectrophotometer at a wave length of 560 mμ using 1.0 cm cells. (Use matched cells). Zero instrument with the zero standard.

8. Plot the optical density values of the standards versus the known concentration (μg/ml) of the standards.

D. Sample Preparation and Analysis

1. Run unknown samples in triplicate.

2. Pipet 10 ml of samples from polyethylene bottles into 25 or 50 ml Erlenmeyer flasks. (Label all flasks correctly)

3. As a blank or zero standard pipet 10 ml of 0.1 M sodium tetrachloromercurate into an Erlenmeyer flask.

4. To each Erlenmeyer flask, pipet in the following order:

a. 1.0 ml of bleached para-rosaniline solution

b. 1.0 ml of 0.2% formaldehyde solution.

5. Allow 25 minutes for color development.

6. Determine optical density reading on a spectrophotometer at a wavelength of 560 mμ using matched 1.0 cm cells. Zero the instrument using the blank solution.

7. From the optical density readings of the unknown samples, determine the concentration in μg/ml from the standard curve.

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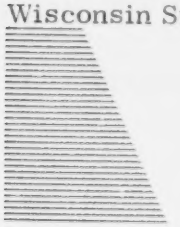

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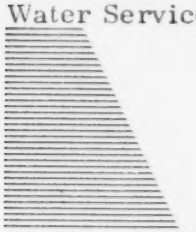

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Sao Paulo - Brasil, Comissao Inter-Municipal de Controle da Poluicao das Aguas E Do Ar
Sydney, Australia, Metropolitan Water, Sewerage, and Drainage Board
Taiwan Institute of Environmental Sanitation, PHA, Taiwan
- Pingtung Air Pollution Laboratory
- Pingtung Organic Waste Laboratory
- Taichung Water Laboratory
- Tainun Water Laboratory
- Taipei Milk Laboratory
- Taipei Radiological Health Laboratory
- Taipei Water Laboratory
- Taitung Water Laboratory

Vancouver B.C., Canada, Department of National Health and Welfare